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HCN@CoS composite electrodes for supercapacitors: microwave-assisted hydrothermal synthesis

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Abstract – The development of efficient, clean and high-performance energy storage devices is urgently needed due to the ever-increasing demand for energy to power products, global warming issues and the depletion of fossil fuels. Transition metal sulfide electrodes play important role in determining electrochemical properties, making them highly promising candidates for supercapacitor applications. The supercapacitor is an effective energy storage and conversion device. It is a sustainable charge storage device that bridges the gap between batteries and conventional capacitors. In this study, CoS material was coated on hollow carbon nanorods (HCNs) with a unique nanorod shapes. Metal sulfide coating on carbon was achieved by microwave-assisted hydrothermal synthesis in 20 min. The resulting samples was characterized by FESEM and EDX. HCN@CoS exihibited a capacitance of 0.45 F cm^{-2} at a current density of 1 mA cm^{-2}. This microwave-assisted synthesis of HCN@CoS electrode provides a promising way to prepare electrodes and expands the utilization of transition metal materials in high-performance energy storage applications.

Keywords – Cos, Supercapacitor, Composite Electrodes, Nanorod Carbon, Microwave Synthesis

I. INTRODUCTION

Energy scarcity and environmental pollution are the most serious problems facing sustainable development. The efficient use of energy and the search for clean and renewable energy sources are promising ways to solve these problems. Therefore, storage technologies, including the synthesis of efficient and low-cost new energy materials and efficient processing methods, are extremely urgent. With the development of science and technology, many types of practical and effective energy storage technologies, such as batteries, fuel cells, electrochemical supercapacitors (SCs), etc., are being investigated [1]. Supercapacitors, also known as electrochemical capacitors or ultracapacitors, have high power density and exceptionally long life compared to batteries. Compared to conventional capacitors, supercapacitors are a type of energy storage device with increased energy density [2]. Carbon-derived materials (graphene oxide, reduced graphene oxide, activated carbon, nanofibres, etc.) are very important in the production of supercapacitor active materials because they have a hollow structure and their important parameters, such as high surface area, pore volume and width, allow ions to easily enter and exit the cavities during the charge-discharge processes of the supercapacitor [3]. Meanwhile, transition metal sulfides such as NiS, MoS_2 and CoS have emerged as attractive electrode materials due to their unique advantages over the corresponding metal oxides [4-7]. Among these, cobalt sulphide (CoS) has a wide range of applications as an efficient electrode material due to its excellent redox activation, high reversible discharge capacity and superior mechanical stability. The aim of this study is to investigate the usability of CoS coated hollow nanorods in supercapacitor applications. HCN@CoS nanoparticles were synthesised using

microwave synthesis method. The microwave synthesis method was chosen because of its fast, energy-efficient and easily controllable synthesis process.

II. Materials and Method

$Co(NO_3)_2.6H_2O$, $Na_2S.9H_2O$, ethanol and KOH were purchased from Sigma-Aldrich. All chemical reagents were of analytical grade and were used without further purification.

A. *Hollow carbon nanorod synthesis (HCN)*

$FeCl_3.6H_2O$ and urea were combined in 50 mL of water and heated at about 90-95°C for about 8 hours. At the end of the reaction, the material obtained was washed three times with deionised water and dried. The product is β-FeOOH. A solution containing 0.63 g of β-FeOOH was added to Tris buffer (10 mM; pH: 8.5) and mixed with dopamine. The mixture was stirred for 24 hours at 50°C. After the reaction, the product was obtained by centrifugation. The material obtained is polydopamine (PDA)-coated iron oxide. To obtain hollow carbon material, the calcination process was carried out in a tube furnace under an Ar atmosphere in two stages: (i) at a heating rate of 1°C/min, the temperature was raised to 400°C for 2 hours, and (ii) at a heating rate of 5°C/min, 500°C was reached for 2 hours. After cooling to room temperature, the product was etched with 2 M HCl to remove the core iron oxide. The final product is hollow carbon nanorods (HCNs) with large voids.

B. *Synthesis of CoS on hollow carbon nanorod (HCN@CoS)*

First, HCNs were coated with PVP polymer. The PVP-coated HCNs were then combined with $Co(NO_3)_2.6H_2O$, hexametylenetetramine (HMT) and trisodium citrate in 50 mL of water. After 8 hours of reaction at approximately 90-95°C, the mixture was cooled to room temperature. $Na_2S.9H_2O$ was added to the mixture and it was placed in the microwave for 20 minutes at 700 W. The final product was obtained after centrifugation and named HCN@CoS.

III. Results and Discussion

A. *Morphological Analysis*

Figure 1 shows scanning electron microscope images of HCN@CoS at various magnifications. As can be seen from the SEM images, CoS grows irregularly on carbon, which is a smooth hollow nanorod with a shape of about 200 nm. The fully carbonised surface is rough and has obvious folds, greatly increasing its specific surface and active area. Furthermore, the coating of carbon nanorods with CoS created a unique nanostructure that could be favourable for electrolyte ion transport.

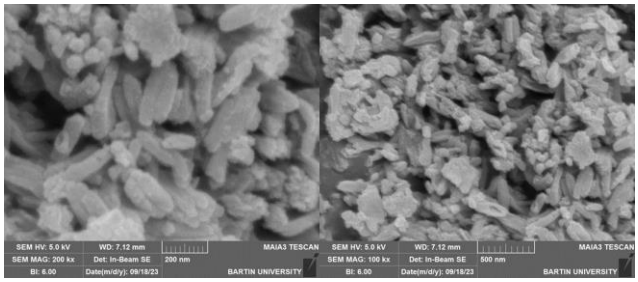

Figure 1. SEM images of HCN@CoS composite materials at different magnifications

In the EDX spectrum of the HCN@CoS structure, only C, Co and S are observed in the structure (Figure 2). The EDX spectrum shows that 7.3 at% is Co and 8.0 at% is S. The fact that the ratio of Co and S in the EDX spectrum is almost 1:1 proves that the material coated on HCN is CoS.

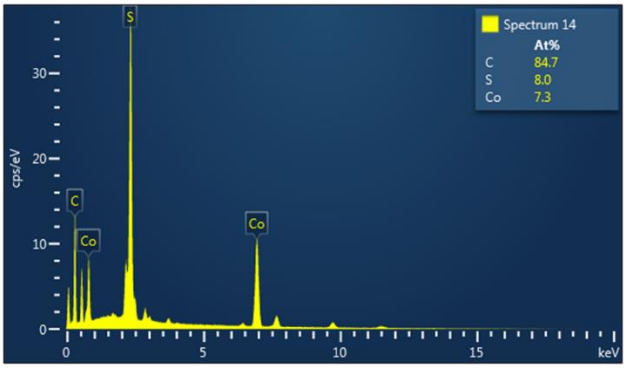

Figure 2. EDX spectrum of HCN@CoS composite material

B. *Electrochemical Analysis*

A three-electrode system with 3 mol L^{-1} KOH as electrolyte, platinum as counterelectrode, Hg/HgO as reference electrode and HCN@CoS as working electrode was set up to obtain the best reaction time of the prepared composites. CV curves of HCN@CoS at different scan rates (10-100 mV s^{-1}) are shown in Figure 3, all CV curves have redox peaks. As the scanning rate rises, the oxidation peak potential grows steadily, whilst the potential of the

reduction peak decreases. The CV curve shows a pronounced deformation, indicating the formation of electrode polarisation at high scan rates.

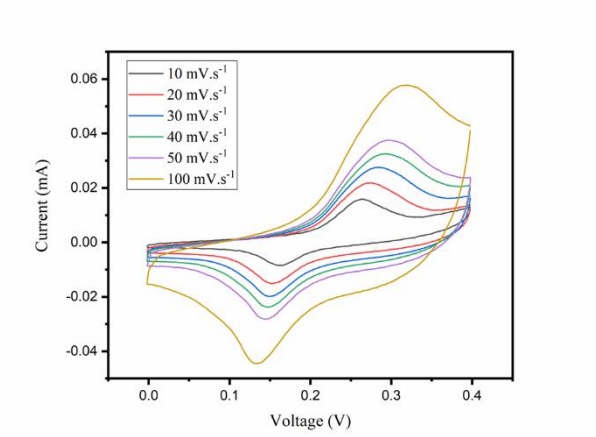

Figure 3. CV curves of HCN@CoS material at different scan rates (10-100 mV s^{-1})

Figure 4 shows the galvanostatic charge-discharge (GCD) plots of HCN@CoS at different current densities ranging from 1 to 10 mA cm^{-2}. The specific capacitance of the HCN@CoS electrode is suitable for use as electrode material in supercapacitors, consistent with the findings from CV measurements.

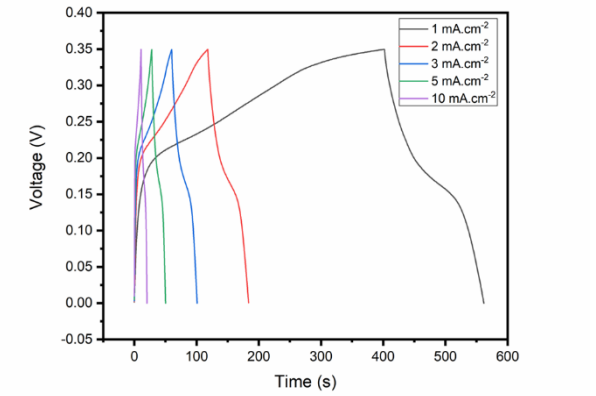

Figure 4. GCD curves of HCN@CoS at various current densities

IV. CONCLUSION

HCN@CoS nanostructures were successfully synthesised using microwave-assisted hydrothermal method. The HCN@CoS electrode offers higher specific capacitance, lower charge transfer resistance and better electrochemical stability due to its barred morphology. HCN@CoS obtained by microwave-assisted hydrothermal synthesis, which is a fast, easy and inexpensive method, provides a capacitance of 0.45 F/cm^2 at a current density of 1 mA/cm^2.The results obtained in these experiments can be an important step for the development of supercapacitor technology.

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